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CONSIDERATIONS FOR THE INSTALLATION OF AN INDUCTIVELY COUPLED PLASMA FOR THE ANALYSIS OF RADIOACTIVE SAMPLES*

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## ABSTRACT

The sensitivity, dynamic range, and sample through-put rate attributes (ref.1,2) of the inductively coupled plasma (ICP) call for its consideration as the instrument of choice for the multielement analysis of radioactive samples. Based on our experience in handling radioactive materials, considerations will be presented concerning safety of the operator, modularity of the ICP-atomic emission spectrometer systems, reduction of the complex actinide spectra, atomization systems, drain and recovery systems, aerosol containment, heat dissipation, radiolysis effects of sample on dry-box environment, and liquid and solid sampling.

## INTRODUCTION

A review by Faires (ref.3) describes many of the attributes of the ICP which make it a valuable tool to be included in the analytical chemist's kit. Its demonstrated reliability makes it economically feasible to consider the ICP for the multielement analysis of radioactive samples in spite of the problems of containment and operator safety. ICP-atomic emission spectrometry (ICP-AES) has found favor in nuclear applications at European installations such as Karlsruhe and Grenoble (ref.4). Homi (ref.5) developed one of the first ICP-AES systems for the multielement analysis of radioactive solutions in the United States. Floyd et al. (ref.6) demonstrated the necessity of separating uranium from the analyte to reduce the interference effects from its complex spectra on the determination of trace elements with ICP-AES. Fassel and Edelson (ref.7) have discussed aerosol containment and heat dissipation.

## OUR EXPERIENCE AND PLANS

Planning concerning glove box design and work must be done carefully. Once the systems are inaugurated and contaminated with radioactive material, analysts may become locked in on methods (because of the expense and safety hazards involved with major modifications) for decades or longer. It is important for the

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planner to consider all possible parameters and options and for the final design to be as flexible as possible.

Operator safety must be considered. The maximum permissible body burden for $^{239}Pu$ is 0.65 μg (ref.8). For samples with β-γ radiation, shielding must be provided. Our glove box plans call for the enclosure to be constructed from a stainless steel - lead - stainless steel sandwich. Operator windows will be of leaded glass. Optical shielding will be incorporated to provide protection from the intense uv radiation of the ICP torch. Protection from rf radiation and electric shock will also be provided. Equipment installed in the box should be easily accessed, easily repaired, and have no sharp edges.

Modularity of instrumentation is dictated by the high cost of ICP-AES equipment. All electronic and optical equipment possible should be kept outside the glove box environment to permit easy and economical repair and to provide protection from radiolysis. Only the torch, nebulizer, spray chamber, and drain system should be located in the glove box.

Reduction of the complex actinide spectra (Fig. 1) must be accomplished by separation of the actinide from the analyte before its introduction to the ICP. This also eases the performance demands on the aerosol containment system.

A block diagram of our conception of an ICP light source glove box is shown (Fig. 2).

Atomization systems should include the capability for routine solution sampling, limited volume sampling for sequential scanning, slurry analysis, and high dissolved solids sampling. A nebulizer - spray chamber - torch system alternative for the analysis of HF stabilized solutions will be included.

Waste radioactive solutions will be recovered from the drain trap by the use of a peristaltic pump to remove excess solution from the drain trap and transfer it to a residue bottle.

Aerosol containment will be accomplished through the use of easily removable filter cartridges. Frequent filter changing will prevent the build up of penetrating radiation in the area of the operator's upper body and head, and will assure free exhaust flow to ease heat dissipation from the ICP torch effluent gas.

Heat dissipation will be accomplished with a heat exchanger situated between the torch and the filter. It should be easily demounted for cleaning and contain an easily replaceable liner.

High α-emitting samples such as $^{238}Pu$ create radiolysis problems by collisions of α-particles with glove box construction materials and environmental gas molecules creating highly excited ions which accelerate corrosion of equipment situated inside the glove box. Chlorinated and fluorinated plastics should be avoided. Even air and water vapor in the box are known to cause problems in the presence of $^{238}Pu$.

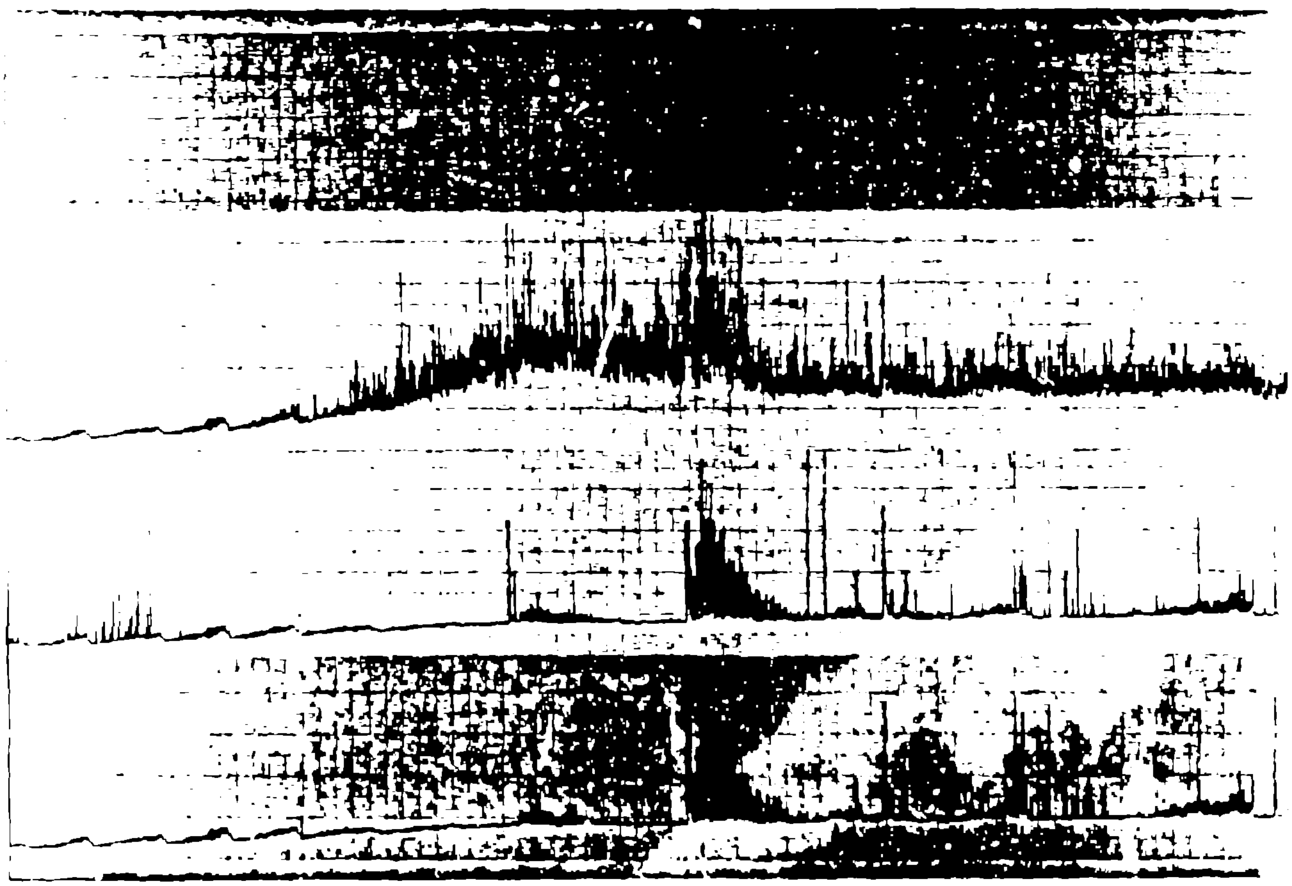

Fig. 1. ICP spectra of: bottom - 0.1 M $HNO_3$, Middle - Cu 10 μg/mℓ, Top - U 100 μg/mℓ. Range 200 (left) to 400 nm (right).

Solid sampling must be considered, as well as liquid sampling, because the majority of samples encountered by emission spectrochemists are solid samples. Apparatus will be included for solid sampling such as laser ablation, slurry handling, and an auxilliary arc capable of high current dc-arc or controlled waveform spark source (CWSS) excitation.

Versatility will be achieved (Fig. 3) by including in the glove box system a second polychromator with exit slits optimized for dc-arc and spark lines and capable of time-resolved recording of the spectra from the auxilliary arc, a balance box, a wet chemistry box, a storage box for contaminated instrumentation and equipment, and a glove box for the development of automated methods such as high pressure liquid chromatography and flow injection analysis.

## CONCLUSION

Careful planning and proper materials selection can lead to a very effective system for the ICP-AES analyses of radioactive solutions and solids. Adequate

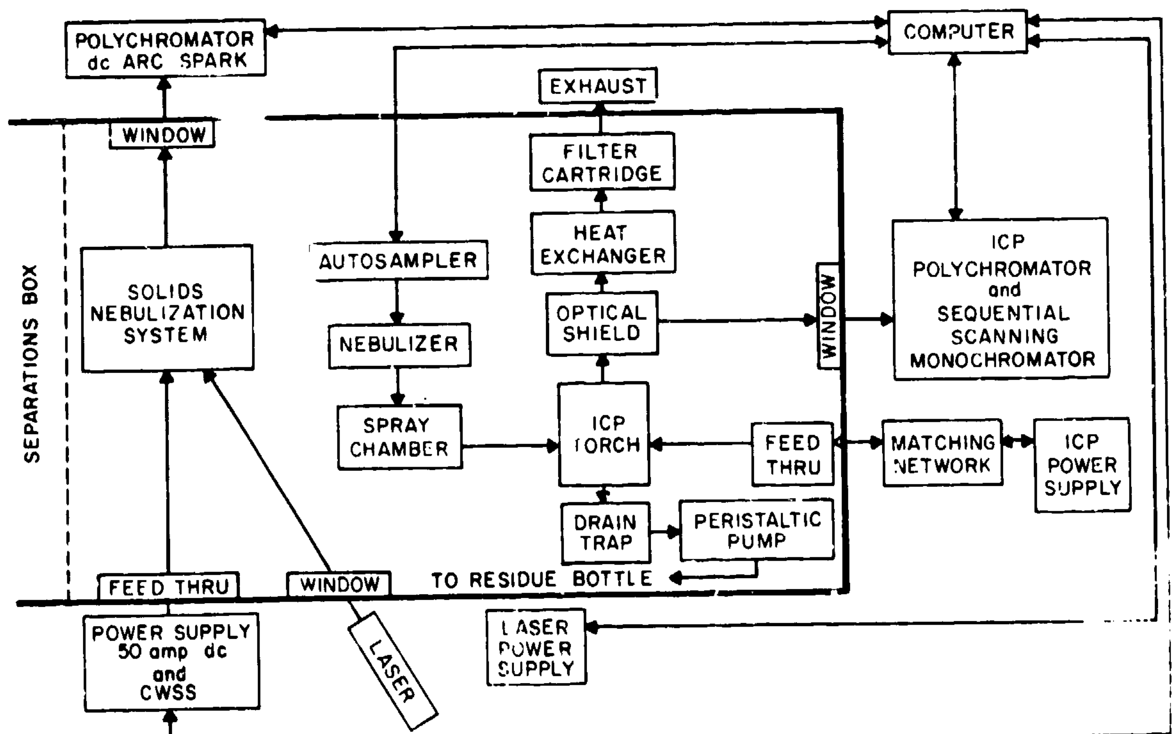


Fig. 2. Light source glove box.

protection can be provided to both the operators and the environment. Finally, careful layout of the glove box train and spectrometer complex can permit the concurrent operations of routine sample analysis and advanced methods development - operations which are usually mutually exclusive.

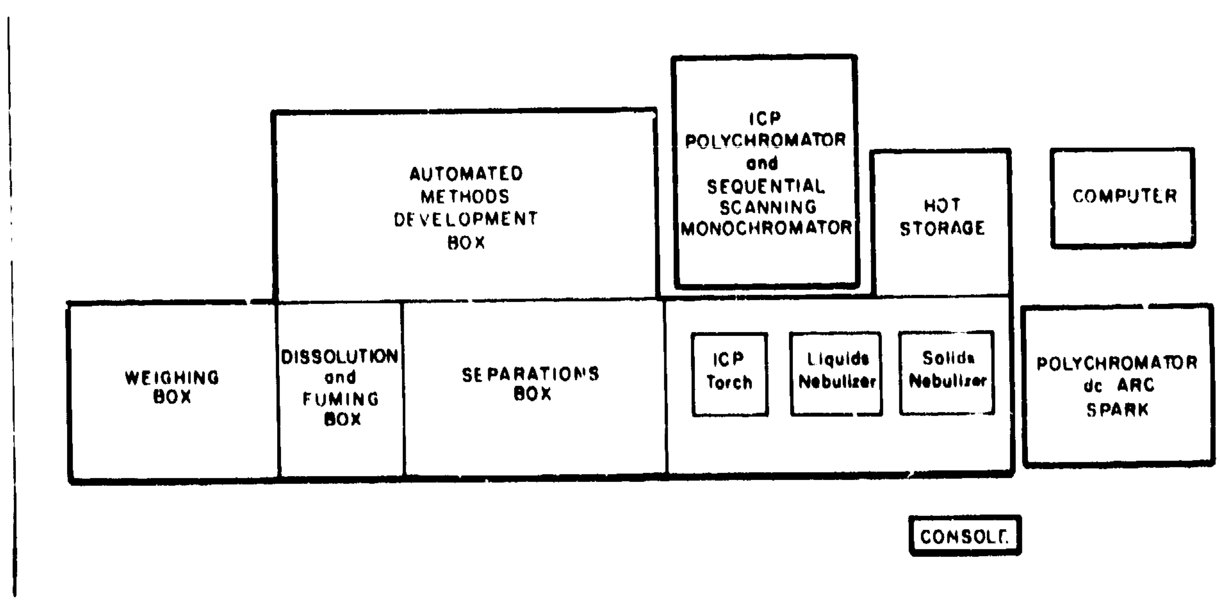


Fig. 3. Equipment and glovebox layout (top view).

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